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SECOND QUARTERLY REPORT

# RESEARCH AND DEVELOPMENT

# IN

# CdS PHOTOVOLTAIC FILM CELLS

By

T. A. Griffin, R. W. Olmsted, J. C. Schaefer

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

August 19, 1964 to November 19, 1964

CONTRACT NAS 3 4177

THE HARSHAW CHEMICAL CO.

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Contract NAS 3-4177

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## FOREWORD

The work of this project has been carried out at the Crystal-Solid State Research Laboratory of The Harshaw Chemical Company. Dr. J. M. McKenzie is the Technical Director of the laboratory. The work was sponsored by the Lewis Research Laboratory of NASA with Dr. Andrew E. Potter acting as Technical Advisor, and Clifford Swartz acting as Project Manager. Project direction has been provided by Mr. J. C. Schaefer. Mr. T. A. Griffin has acted as the Project Supervision Engineer and Principal Investigator.

The following Harshaw Chemical Company personnel have contributed to this program: R. W. Olmsted, W. W. Baldauf, D. H. Dickey, D. J. Krus, and R. Rautenstrauch.

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# SUMMARY

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During the period covered by this report, improvements in cell power-weight ratio, and overall efficiency have been realized. Power-weight ratios of forty watts per pound and efficiencies of 3.5% have been observed consistently.

Different methods of plating grids and barriers have been examined. The more promising of three methods have been pursued with the intent of increasing cell efficiency.

Film and barrier properties were examined with the objective of producing them with a more consistent uniformity. Structure and arrangement of CdS crystals within the film were examined microscopically.

Various substrate materials were tried in an effort to find one lighter and more compatible with CdS films. Molybdenum remains the best to date. It was determined that CdS could be plated on conducting paper. This will be investigated further in the next quarter.

Cell fabrication techniques were examined in order to improve cell efficiency and reduce fabrication time. Drawings for an impulse laminator were obtained and arrangements made for procurement.

RESEARCH AND DEVELOPMENT

IN

CdS PHOTOVOLTAIC FILM CELLS

## INTRODUCTION

This report covers the second quarter of work on contract NAS 3-4177 which is a follow-on contract to NAS 3-2493. The present work places emphasis on making the film cell more uniform and reliable under launch and space conditions. The major objectives are tc fabricate light weight, flexible, uniformly high efficient, low cost cells capable of operating in space conditions for long periods of time.

This program will be implemented by a performance in-house test program at NASA's Lewis Laboratory. The results of tests such as ultra-violet exposure, electron and proton bombardment, and thermal cycling in high vacuum will be used to modify cell materials and construction in order to find those combinations that are most compatible with prolonged operation under space conditions.

During the first quarter the CdS thin film cells had been improved in power to weight ratio to about 40 watts per pound for 3" x 3" cells. This represents a significant increase over the 25 watts per pound reported on the previous contract.

Electroplated grids have produced 3% cells. Although 3% is below the average cell efficiency there has been a constant improvement toward the higher value. Cells of this type construction promise to reduce the cost by elimination of the costly gold electroformed mesh. The new cell packaging design has withstood hundreds of thermal cycles conducted in a vacuum chamber. The temperature cycles were conducted between +65°C and -70°C.

The procedure for applying the electroplated collector grid has been found to have a deleterious effect on the cell. The effect is in proportion to the total plating time. Plating conditions have been altered to provide a shorter deposition time and consequently improved cells. Auxiliary bus bars have been attached to the electroplated grids and have been found to improve the cell output. Other types of plating baths are being investigated.

Investigation of substrates other than molybdenum has been continued. The interface problem associated with Invar has been defined. Indications of non-uniformity in the barrier layer have been noted.

The pilot line has been converted to the new chemiplated type barrier. This change has resulted in an increase from an average of 2.6% to an average value of 3.5% for cell conversion efficiency. The highest value reported for this period is 5.1%. An array of 4 - 3" x 3" cells was delivered to the contract monitor with a power to weight ratio of 30 watts per pound.

## ELECTROPLATED GRIDS

An early method for current collection of the Cadmium Sulfide (CdS) cell involved hand ruling the barrier surface with a silver paste or ink. Much of the active surface was covered and the conductivity of the silver conductors was low.

The introduction of a high purity gold metal electroformed collector with a light transmission range of 83% to 87% was a milestone in the collection of the power generated by a given cell. The gain in collector efficiency was at an increase in cost. The gold electroformed grids were expensive.

In order to evaluate a cell with the electroformed grid, the cell had to be laminated. This required considerable time until a test fixture was designed that permitted quick and economical cell evaluation. This device permitted intimate contact between cell and collector during the test period but before actual lamination. This fixture has been adopted as standard equipment.[1]

Such a procedure was satisfactory at this stage of development. Two hundred and sixty cells were delivered to NASA, Lewis Research Center, Cleveland, Ohio, for an in-house testing program. One of the tests required repeated thermal cycling to simulate orbital flight. The early cells delivered for this purpose were constructed with the electroformed gold collector electrode. Many cell units survived 400 cycles and then became erratic in behavior. Four hundred cycles were considerably less than the desired one year orbital equivalent.

A change in collector electrode design was indicated. Experimental investigation resulted in a procedure for the electrodeposition of a pure gold grid on the surface of the cell barrier. Very low yield and relatively low power cells resulted. The operating cells of over 2.3% conversion efficiency were placed on test to determine whether this design was worthy of further work. This type cell was found to be capable of exceeding 1200 thermal cycles. Additional work has been performed in this area concentrating on the causes for the low efficiencies since the cells were invariably 30% higher when initially tested. One factor that appeared to be contributing to the low output was a lack of sufficient conductor cross section. Total plating time (2 hours) and increased plating current densities increased the grid thickness. However, the long period of immersion was suspected as the major cause of the power loss. This was confirmed when it was found that the cells improved as the plating time was reduced.

Several cells have been made with the electrodeposited gold grids and have been found to develop the same power and efficiency as indicated by the pressure contact test.

Efforts will be made to further reduce the time of plating.

1. Gold Plating Process

The gold plating process initially required 0.45 ma/cm$^2$ current density, 21$^\circ$C bath temperature, and a plating time of two hours to obtain a gold thickness of 1.2 microns. Later, bus bars were plated on the barrier along with the grid pattern in an attempt to increase conductivity. Since the total current flowing through the bath could not be increased, the current density dropped to 0.3 ma/cm$^2$. The lower current density produced an average conductor thickness of 0.8 microns.

At this point it was obvious that the information obtainable from the handbooks and manufacturers literature on these baths was not adequate to solve the plating problem. A study program was needed that would produce data concerning all parameters in a relatively short period of time.

a. The Hull Cell

A standard test cell used by electroplaters to determine plating conditions is known as the Hull Cell[2]. This device has proven to be of immense value for research. In use the cell is filled to an exact level with the plating solution in question and an anode is placed at the rear of the cell. The cathode is inserted opposite to the anode, but is placed at a prescribed angle. A specified current is caused to flow. The net effect of the angle is the non-uniform distribution of current across the face of the cathode. The end nearest the anode has the highest current density while the other end has the lowest current density. This gradient permits the selection of the desired deposit. Repeated tests at various currents, temperatures and current density range provides data rapidly.

Both nickel-coated brass cathodes and solar cell cathodes were used in the Hull cell in order to evaluate plating current densities and temperatures. The results, which were obtained by visually comparing the cathodes, indicated that a temperature range of 21$^\circ$C to 66$^\circ$C and a current density range of $\triangleright$ 0 to 2 ma/cm$^2$ would yield a uniform, non-pitted deposit.

Grids were electroplated onto solar cells at four current densities (0.3, 0.6, 1.0 and 1.5 ma/cm$^2$) and three temperatures (21, 49 and 66$^\circ$C). The time in the solution for each set of variables were adjusted so as to give grid thickness of about two microns. Optimum plating conditions were determined on the basis of three cell characteristics; differences in $I_{sc}$, $V_{oc}$ and the IV curve shape before and after grid electroplating. The plating conditions which resulted in the smallest changes in the cell were found to be 1.0 ma/cm$^2$ current density at 49$^\circ$C and a plating time of 45 minutes. These conditions produced a grid thickness of two microns. Under these conditions, cell degradation caused by plating was still observed although the amount of efficiency loss was considerably decreased.

Experiments are now in progress to determine whether an initial thin layer of gold applied at a low current density, followed by an additional thick layer deposited quickly at a high current density will further reduce the degradation. Ideally, this method could reduce the plating time to ten minutes.

b. Effects of Light During Plating

Light could conceivably influence the cell during the plating process since the bulk CdS is photoconductive in nature and the barrier degradation is believed to be caused by a photochemical process. Experiments conducted to evaluate the effect of light on the cell during grid electroplating have shown no observable effects. These experiments will be repeated when the optimum electroplating techniques have been established.

2. Other Plating Methods

a. Aqueous Solutions

Cells gridded by the electrodeposition of copper and nickel from aqueous solution have been found to be short circuited.

Electroless depositions of nickel were made on several cells. Two different solutions were used with both solutions depositing nickel on the grid lines and on the photoresist. Cell degradation and difficulty experienced with the removal of nickel covering the photoresist resulted in a rejection of this approach.

b. Solutions with Alcohol as the Solvent

Solutions were made in which alcohol rather than water was utilized as the solvent. Experiments were conducted with two low-water content electrolytic solutions: (1) copper sulfate in methyl alcohol and sulfuric acid, and (2) silver nitrate in methyl alcohol and nitric acid. The copper solution gave good, heavy uniform deposits, but resulted in shorted cells. The silver solution was not successful since the silver nitrate would not remain in solution.

An immersion process consisting of a solution of gold chloride and methyl alcohol was studied. The mechanism involved a reaction which continued only until the surface was lightly plated. These tests also produced degraded cells.

Many plating techniques were found in the literature but initial considerations eliminated these as incompatible with the CdS-$Cu_xS_x$ system. Continued efforts in this area will be concerned mainly with low water content solutions.

3. Auxiliary Bus Bars

A photoresist mask has been designed which enables gold bus bars to be plated simultaneously with the grid. Presently the thinness of the gold bus bars, and its resultant resistance, necessitates spot welding silver foil leads on top of the gold. Efforts are being made to increase the thickness of the electroplated bus bars by using different anode configurations. The value of the auxiliary bus bars will be discussed in the section on lead attachment.

## FILM PROPERTIES

Early CdS evaporated films were produced using single crystal chips as the evaporant. Later it was determined that a sintered CdS product was sufficient for the purpose. Rather recently it was discovered that the CdS powder as supplied by the manufacturer was adequate. Several manufacturers' products were examined. All but one were found to be unsatisfactory because of the range in particle size. The General Electric powder meets the particular requirements for the fixtures used in the present evaporators because no particles range below one micron.(3)

The presence of fines in the evaporant has caused spattering of the substrate. One of the powders compared with the General Electric CdS powder was a Sylvania CdS powder which by spectrographic analysis, was only slightly more contaminated than the General Electric material.(4) It also contained many fines and could not be used without redesigning the filaments.

Recently samples were obtained of a higher purity Sylvania CdS and tested. This material was found to have a more uniform particle distribution. Table I is a spectrographic comparison of samples of General Electric and Sylvania material. The two are very similar in content for the impurities determined in the analysis. Films were made from both General Electric and Sylvania powders under normal conditions. The resulting films were very different. The films from the Sylvania material were powdery but without evidence of spattering. They also had a yellow layer next to the substrate. The General Electric powder produced the type of film normally expected.

Two approaches were tried to make usable films from the Sylvania powder. Films formed under the usual evaporation conditions were heated to 600$^{\circ}$C in argon for one hour. Much of the powdery appearance disappeared but the molybdenum substrate was also altered. It appeared that a molybdenum oxide was being formed, indicating a possible air leak in the furnace. These tests will be repeated under oxygen-free conditions.

The second method employed the deposition of Sylvania material on the substrate at a temperature 100$^{\circ}$C higher than normal. The films that were produced appeared similar to the films made with standard material at the standard substrate temperature.

The following is a comparison of the properties of typical films produced from the two types of materials.

| | Sample N94 (Sylvania) | Sample N96 (G.E.) |
|---|---|---|
| Thickness | $1.2 \times 10^{-3}$ cm | $2.4 \times 10^{-3}$ cm |
| Hall Coefficient | 7.47 | 2.76 |
| Mobility | 16.3 cm$^2$/volt-second | 0.79 cm$^2$/volt-second |
| Carrier Conc. | $8.37 \times 10^{17}$ cm$^{-3}$ | $2.26 \times 10^{18}$ cm$^{-3}$ |
| Resistivity | 0.46 ohm-cm | 3.5 ohm-cm |

TABLE I

QUALITATIVE SPECTROGRAPHIC ANALYSIS

OF CdS POWDERS

| | G.E. CdS Powder (Lot 185) | Sylvania CdS Powder (Lot T-32-1) |
|---|---|---|
| Aluminum | FT | $FT^-$ |
| Calcium | VFT | VFT |
| Copper | VFT | $VFT^+$ |
| Indium | --- | --- |
| Iron | $VFT^+$ | $VFT^+$ |
| Magnesium | VFT | VFT |
| Manganese | --- | --- |
| Nickel | --- | --- |
| Silicon | VFT | FT |
| Thallium | --- | --- |

Key:
T = Trace .001-.01%
FT = Faint Trace
VFT = Very Faint Trace
--- = Not Detected

The Sylvania films formed at the higher substrate temperature had a lower resistivity. Normally, a high substrate temperature will eliminate much of the free cadmium in the CdS film resulting in a resistivity increase. Neither film was doped.

Cells were made from films of both materials. While the cells from the Sylvania material did not develop the same power as the G.E. material, it is possible that with certain adjustments equivalent cells could be made. The Sylvania cells were found to be about 75% as efficient as the G.E. cells: This might be attributed to the low resistivity in the Sylvania films as indicated by the poor shunt characteristics of the I-V curves. More work with the Sylvania material is indicated.

## ALTERNATE SUBSTRATE MATERIALS

Since the discovery of the chemical milling process, the use of thinner molybdenum foils has not been pursued. A lesser handling problem is presented if thicker foils are used for cell fabrication and as a final step the back of the molybdenum substrate is chemically milled to the desired thickness.

Investigation of other metallic substrates that appear more desirable than molybdenum because of cost, density, conductivity, thermal expansion or ability to form a good contact to n-type CdS has been made in the past as a means of achieving weight reduction. At times, the results have been promising, but none have yielded the reproducibility achieved with molybdenum.

Several of the metals investigated have been found to cause a yellow powdery layer to form immediately adjacent to the substrate. While working with the high purity Sylvania CdS it was discovered that the yellow powdery layer can be eliminated by employing very high substrate temperatures. In the past, Invar had been known for formation of the undesirable intermediate layer. Consequently, some Invar was tried as a substrate at elevated temperatures using the standard CdS material. As in the case of the Sylvania material on molybdenum, the yellow layer disappeared. The adherence of the CdS to the Invar also seemed improved. This higher substrate temperature may be the key to getting consistently good cells on Invar. In previous work, cells made on Invar have been found to be very good or very poor with none of intermediate quality.

Films were evaporated onto several substrate materials during this quarter. Tin clad steel was one of the new materials investigated. Adhesion of the CdS was poor and curling was severe. These difficulties were caused by substrate surface condition and mismatch of thermal expansion coefficients. Good CdS films have been produced on a conductive paper. So far, cells have not been fabricated from this material.

Experiments were also performed to determine the cause of the degradation which has been noticed with cells fabricated on titanium substrates. A cell was chosen that degraded about 50% from the original value. The CdS film was stripped from the cell. The surface of the titanium was found to be a very dark grey. In an attempt to determine the cause of the degradation, pieces of polished titanium were packed in sulfur and in CdS powder. These samples were heated to 150°C in argon and then allowed to remain in the closed container for two days. In both cases, the titanium surface turned grey. The samples were not as dark as the titanium used as a substrate. These tests will be repeated for a longer period of time.

## LEAD ATTACHMENT

The attachment of the silver leads to the electroplated grid is an area that has required much attention.

In the first quarter, several tests on the effect of solder on the barriers were reported.[5] The conclusions were that the indium type solder will attack both the electroplated and the chemiplated barriers if the amount of solder is not carefully controlled. The chemiplated barrier seemed most sensitive to the solder. All of these tests were conducted on cell packages fabricated with the one mil thick electroformed meshes.

When the electroformed mesh was replaced by the very thin, two micron thick, electroplated grid, the solder problem arose again. It was nearly impossible to apply sufficient solder to attach the auxiliary silver bus bars and leads to the collector while at the same time using a minimal amount to prevent attack on the barrier. A thicker grid could alleviate the problem. This is being attempted as reported in an earlier section of this report.

Successful cells have been fabricated by initially depositing gold bus bars as previously described. An auxiliary silver bus bar and lead is spot welded to the top of this gold stripe. A micro-gap welder has been purchased by The Harshaw Chemical Company for this purpose. A unit of this type prevents damage to the cell barrier during welding. Figures 1 through 4 demonstrate the improvement in cell efficiency by means of the welded auxiliary bus bars and leads. These figures are a comparison of two cells before and after gridding. The I-V curves were taken under 100 mw/cm$^2$ equivalent sunlight. Figure 1 is the I-V curve of cell L88-D before gridding. Figure 2 is the I-V curve after gridding and attachment of the silver leads. Cell N79-D was fabricated in the same manner with one exception. The silver bus and leads were not welded but were held in place by pressure applied during encapsulation. Cell L88-D was 0.8% efficient and Cell L79D was 0.9% initially. (Figures 1 and 3). After gridding Cell L88D was 2.7% efficient (Figure 2) while Cell L79-D was only 2.3% efficient (Figure 4). These results are typical.

Perhaps more important than the initial improvement in efficiency for cells with welded connections over those with pressure connections is the improvement in the cell package with regard to thermal cycling. The indications from tests run here and those in progress at Lewis Laboratory, NASA, are that the welded connection greatly improves the reliability and life of the cells during repeated thermal cycling in high vacuum. From

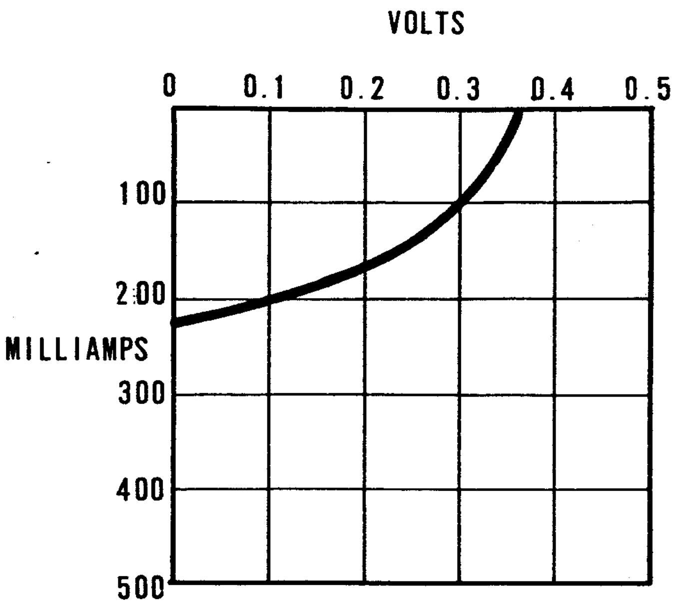


Figure 1

I-V Curve of Cell L88-D in Pressure Test Unit

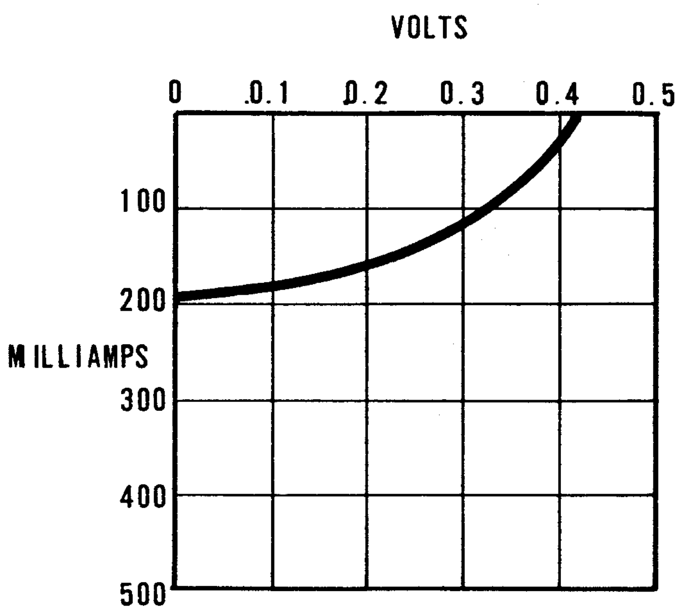


Figure 3

I-V Curve of Cell N79-D in Pressure Test Unit

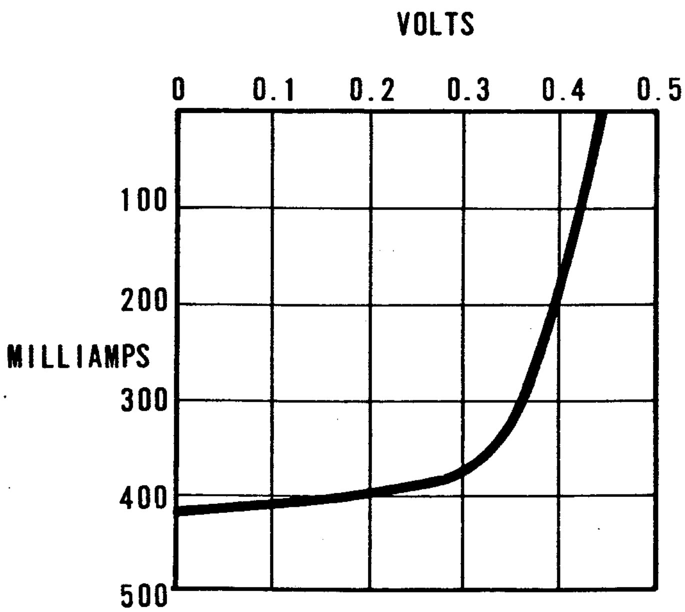


Figure 2

I-V Curve of Cell L88-D with Welded Bus Bars

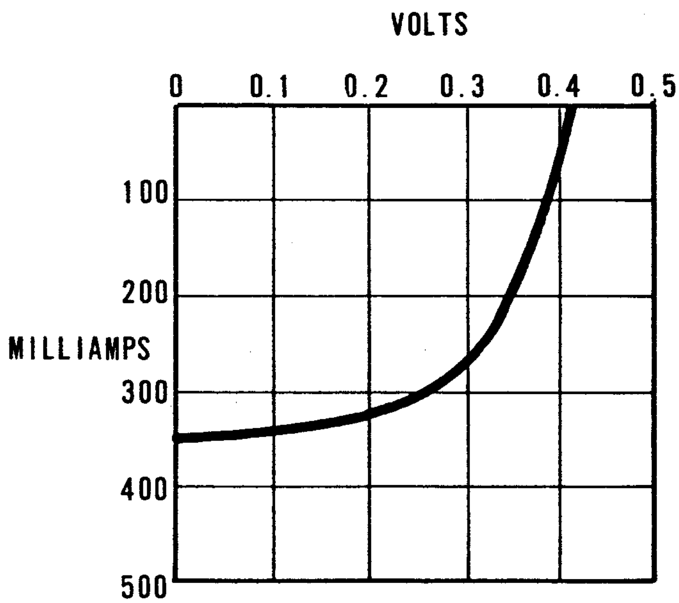


Figure 4

I-V Curve of Cell N79-D with Pressure Contacted Bus Bars preliminary results, it appears that this type of collector arrangement is more satisfactory than any previous type. When the tests are completed, it is anticipated that any indicated improvements will be incorporated in the cell package.

## IMPULSE LAMINATOR

The present laminating procedure requires that the cell package be placed in platens and heated in a laminating press for more than one-half hour. Since the plastics and the cells have properties that change due to heat, it is desirable to minimize this heating period. It has been proven by experiment that this can be accomplished by using an impulse type laminator. The drawings of an impulse laminator capable of completing the encapsulation cycle in a few minutes have been sent out for bids. This unit should be in operation before the end of the next quarter. It will be capable of laminating up to one foot square arrays.

## FILM AND BARRIER UNIFORMITY

It has been noted in the past that cell geometry appears to be related to conversion efficiency. Nominal 3" x 3" cells have displayed a tendency to have higher efficiency than 1" x 3" cells. On large area cells, it has been found possible to locate small areas with efficiencies higher or lower than those of the large areas. A complete theoretical explanation of these observations has not been found. In analyzing the problem, it is necessary to determine the uniformity of the CdS film and the barrier layer. Better uniformity should provide higher cell efficiencies.

### 1. Film Uniformity

CdS evaporated films exhibit certain evident non-uniformities while probably possessing others not as yet identified. Non-uniformities already observed are of a visual nature. Films differ in appearance from one evaporation to another and even different parts of individual cells may not appear uniform. One difference is in color. Skilled technicians have reported this difference and have predicted fairly accurately on this basis which films will produce good cells. These films are said to be produced with slightly different substrate temperatures. These indications of difference have not been confirmed spectrophotometrically.

Another non-uniformity observed in the CdS evaporated films is the texture. Some areas show an imperfect specular appearance. Other areas have a velvety reflectance, perhaps due to crystal growth facets of approximately the same orientation.

Under microscopic examination, five types of surface formations have been observed. The first type can be characterized by a nearly flat surface. When this surface covers a large enough area, it appears somewhat specular to the unaided eye. The second type is a pit with the suggestion of geometrical regularity in its form. When the population density of such pits is high, the area has a velvety appearance. The third type is a formation caused by a particle of CdS loosely bound to the film. A sprinkling of these particles on the film is referred to as "spatter." Frequently, an amorphous pit is found beneath the particle, suggesting that the particle was deposited before the full thickness of the film was built up. The fourth type appears as a shapeless bump or protrusion in the surface of the film. This type of bump may be an adherent particle of CdS which has been deposited as spatter at an early stage of the film formation and becomes subsequently covered as the film continues to develop. Under high magnification positive discrimination between such a bump and the pit mentioned above is difficult. The fifth type of formation is referred to as a "pinhole." It is a narrow perforation which usually extends through the film to the substrate. Sometimes the form of the pinhole suggests that it is in the same family as the regularly shaped pit mentioned above. Another instance may look like the pit remaining when a CdS particle has broken away from the film. Certain pinholes can't be identified as belonging to either class. Pinholes have been found in cells which were "shorted."

In films where surface texture appears the same to the unaided eye, a difference in color seems to be related to a microscopic difference in texture, and not of type. The surfaces in both cases are flat with a sprinkling of microscopic pits.

Both film color and texture are probably influenced by variations in evaporation rate and substrate temperature, from film to film and from area to area on an individual film.

2. Barrier Uniformity

Various methods of testing cells for barrier uniformity have been investigated briefly. Some non-uniformities have been noted which seem to effect the electrical properties of the cells. The methods and observations are outlined below.

Some cells with low short-circuit currents show a sprinkling of spots where the "barrier" was not formed. These spots have been observed under a microscope with polarized light at normal incidence. This suggests the use of polarized light in other types of inspection such as Brewster angle observations.

Thermoelectric probing was tried but without useful results. For the CdS cells, special equipment and techniques probably would need to be developed for this method. It would require a heated probe made of a material with a Seebeck coefficient approximately the same as the barrier.

Occasionally, a cell with an electroplated barrier will show very noticeable variations in visual appearance across the surface of the cell. That is not always an indication of the cell's efficiency. A few of the low efficiency ones were examined. Bluish areas displayed ordinary I-V curves and were responsive to red light. Reddish areas of the barriers displayed various unusual I-V curves and were responsive to blue light. It is still too early to make any conclusions concerning these tests.

Further optical observations will be made and compared with fabrication parameters and cell operation. A four-point probe resistivity test unit (A. M. Fell, Ltd.) and the method of analysis described by L. J. Swartzendruber(7), may provide a means of estimating the uniformity of the barrier sheet resistivity. It is planned to obtain electron probe data for additional indication of barrier uniformity.

## PILOT LINE

During this period, the contamination in the transistor washer was eliminated. The efficiency of the electroplated cells has gradually increased until it is now back to normal. One hundred sixteen cells were fabricated on the standard line this quarter. The highest electroplated cell was 3.2% while the average was 2.6%.

At the beginning of the third month of this quarter, it was decided to convert the standard line from electroplated cells to chemiplated cells.

The chemiplated cells in general do not show the consistency that the electroplated cells have shown as far as resistance to degradation caused by moisture. However, individual chemiplated cells have shown superior resistance to degradation. The main consideration in switching to the chemiplated barrier was the higher level of efficiency. The electroplated cells were operative at a level of 2% to 3.5% while the chemiplated cells range from 3% to over 5%.

Only 12 cells were done in this manner. The average efficiency was 3.53% and the highest was 5.1%.

During the quarter, one array of four three-inch by three-inch cells was delivered to the contract monitor. This array was over 3% efficient and demonstrated over thirty watts per pound. It is expected that by the end of the contract period, goals of fifty watts per pound for individual cells and forty watts per pound for arrays will be attained.

## WORK PLANNED FOR THE NEXT QUARTER

1. Work will be continued on the electroplated grid. Shortening of the plating time and use of non-aqueous solutions will be the areas of concentration.

2. The impulse laminator will be assembled and put into operation.

3. Work will be initiated on sintered CdS on substrates.

4. Other substrate materials will continue to be investigated. Emphasis will be on Invar at the high substrate temperatures.

5. The study of the uniformity of the films and barriers will continue.

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